



## Research article

## Effects of biochar on the transformation of cadmium fractions in alkaline soil

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## ABSTRACT

To investigate the chemical properties in the biochar-mediated transformation of soil cadmium (Cd) fractions, the effects of biochar applied at different pyrolysis temperatures on soil Cd-fractions, pH value, and soil organic matter (SOM) were studied through an in-lab incubation experiment on contaminated soil. The results showed that the dissolved organic carbon (DOC) of CsBC300 (biochar prepared at 300 °C) was significantly higher (up to 1.31 times) than that of CsBC600 (biochar prepared at 600 °C). However, CsBC600 was more aromatic. Due to the difference in pyrolysis temperatures, the Cd deactivation mechanism of CsBC300 and CsBC600 was mainly to provide a large amount of organic matter and aromatic functional groups to the soil, respectively. The addition of these two biochar types significantly reduced the acid-extracted Cd content, by 76.56–83.52% and 70.48–76.81%, respectively. Contrastingly, it increased the residual Cd content by 2.26–2.36 and 2.08–2.29 times, respectively, which promoted the Cd transformation from the unstable to the stable state. However, CsBC300 had slightly better deactivation effect than CsBC600 on the 120th day, which was due to the decrease of soil pH and the increased SOM content. These study results can provide a theoretical reference for the remediation of Cd-contaminated alkaline soil.

## 1. Introduction

Soil is a medium connecting organisms and non-organisms, while also being a “sink” of environmental pollutants. With the development of the global economy, especially the expanding scale of mining, smelting, agriculture, and modern industry in the developing countries, heavy metal pollution has become a major global problem. According to Bulletin of the National Survey of Soil Pollution in the 2014, the exceeding rate of pollutant points in the cultivated soil was 19.4% in China, with the most serious heavy metal pollution being Cd, which even exceeds the standard rate by up to 7%. Heavy metals were reportedly the main pollutants which affected the agricultural soil quality, with Cd being the primary pollutant as per the Bulletin of China’s Ecological Environment in China 2021. Cd is one of the non-essential elements with the highest toxicity and carcinogenicity. Additionally, it has strong mobility in the soil, could easily be absorbed by crops, and easily bioaccumulated in the human body through the food chain, thereby threatening human health [1]. The ultimate purpose for treating Cd-contaminated soil is to reduce the Cd content in crops, thereby reducing its

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human intake. Currently, the *in-situ* repair with restorative agents has been widely studied and applied for its low cost and simple operation [2-4]. Among the multiple restorative agents, biochar had attracted extensive attention from researchers worldwide due to its great application potential in the adsorption and fixation of heavy metal Cd [5-7].

Numerous studies have reported the Cd adsorption capacity of biochar in environmental media. Regardless of the Cd<sup>2+</sup> presence in water or in soil, biochar has high adsorption capacity through functional groups, physical adsorption, and electrostatic attraction, with the biochar modification also having extensive research prospects [8-11]. As a good adsorbent and soil conditioner, biochar can not only directly adsorb and bind heavy metals through surface functional groups [12,13], but also affect Cd fractions by changing both the soil physicochemical properties and microbial community structure [14-16]. For example, [17] showed that coconut shell biochar significantly reduced the available Cd content in soil, improved soil urease activity, and increased the relative abundance of soil microbial community. According to previous studies, *Pennisetum sinense* Roxb biochar and coffee grounds biochar also reduced the Cd-induced toxicity by increasing the soil pH and enhancing the soil catalase, urease, and phosphatase activities [18,19]. Qiu et al. [20] found that the *Spartina alterniflora* biochar that prepared at 450 °C could effectively reduce the total exchangeable soil Cd by 24%. Jiang et al. [21] studied the restoration of Pb and Cd contaminated sites using litchi branch biochar near the mine. They found that litchi biochar could significantly improve the overall soil properties (pH, organic matter, and Cation exchange capacity (CEC)), while also reducing the Pb and Cd accumulation in crops. Therefore, these results indicated that biochar could affect the fraction transformation of heavy metals and modify the soil properties.

For biochar, the different preparation temperatures can change its physicochemical properties, which then affect the soil properties. For example, the biochar CEC content produced by the pyrolysis of wheat straw at 350 °C, 450 °C, 550 °C, and 650 °C first increased and then decreased with the increase in temperature. The biochar prepared at 350 °C has the lowest CEC content (~15.3 cmol/kg), while those prepared at 550 °C had the highest content (~43.4 cmol/kg), with the O/C and H/C ratios continue to decline [22-24]. This is also the basis for the difference of functional groups between the low-temperature- and high-temperature-generated biochars. It has also been found that with the increase in the preparation temperature, the biochar CEC content change with the change of preparation temperature range, and its directionality is uncertain, which may be caused by different biomass materials [25]. Yang et al. [26] found that when the pyrolysis temperature was 450 °C, the porosity of the rice straw biochar and rape straw biochar were the highest. Here, the phenomenon that the increasing preparation temperature increases the biochar pH is consistent. The specific surface area of biochar is also easily changed by the oxidation and pyrolysis conditions, which consequently affects the other biochar characteristics [22-24]. Pyrolysis temperature confers biochar with certain characteristics. The addition of biochar derived from corn straw and rice straw in corn field and rice field, respectively, increased the soil pH by 3.01 and 2.51 units, while the soil CEC content and organic carbon content increase significantly [22-24]. Therefore, the addition of biochar prepared at different temperatures is the reason for the change of soil's physicochemical properties, including cation content, water holding capacity, and pH value.

However, currently, the remediation of Cd pollution mainly focused on the acidic soils and rice crops in southern China, with very few reports focusing on the remediation of Cd-contaminated alkaline soils. Therefore, the objective of this study was to investigate how the chestnut shell-derived biochar with different preparation temperatures affect not only the availability and fractionation of Cd, but also the soil's physicochemical properties in Cd-contaminated alkaline soil.

## 2. Materials and method

### 2.1. Materials

The studied soil was collected from the alkaline wheat field around a coal gangue hill in the Shandong Province (surface layer 0–20 cm). Then, samples were air-dried, passed through a 2 mm sieve, and mixed homogeneously. The properties of the initial soils were: pH was 9.01, SOM was 27.08 g kg<sup>-1</sup>, total nitrogen (TN) was 2.11 g kg<sup>-1</sup>, total phosphorus (TP) was 0.35 g kg<sup>-1</sup>, total kalium (TK) was 8.32 g kg<sup>-1</sup>, total Cd was 2.10 mg kg<sup>-1</sup>, total Zn was 111.99 mg kg<sup>-1</sup>, total Pb was 86.17 mg kg<sup>-1</sup>, total Ni was 45.48 mg kg<sup>-1</sup>, and total Cr was 85.95 mg kg<sup>-1</sup>. According to the Soil Environmental Quality Risk Control Standard for Soil Contamination of Agricultural Land (GB 15618-2018, China), this sample is designated as the alkaline medium Cd-contaminated soil.

The chestnut shells were collected from the farm around the Qufu Normal University. These were then rinsed thoroughly, dried at 60 °C for about 48 h and finally chopped into small parts. These were then filled in magnetic boats (~10 × 7 × 7 cm), heated separately to 300 °C and 600 °C in an electric tube furnace under oxygen limiting conditions for 2 h with a heating rate of 5 °C min<sup>-1</sup>. After cooling both these to room temperature, the prepared biochar was finely ground and sieved to pass through a 2 mm sieve, thoroughly mixed, and were named CsBC300 and CsBC600, respectively.

### 2.2. Experimental method

First, 1 kg air-dried Cd-contaminated soil was distributed into PVC basins with three replicates, along with 1.0%, 3.0%, 5.0% and 7.0% of biochar addition and being mixed thoroughly. These were then incubated for 120 days in ambient temperature (25 ± 1 °C) and 60% field capacity was maintained by adding distilled water. Soil samples were periodically collected on 0, 5, 10, 20, 30, 50, 70, 90, and 120 days to determine their physicochemical properties and chemical fractions of Cd.

### 2.3. Samples testing

Biochar characterization: Elemental composition was determined using the elemental analyzer (Elementar Analysen Systeme

GmbH, Hanau, Germany). The specific surface area sizes were determined by the specific surface area analyzer (Kubo-x1000, Beijing Pyod Electronic Technology Co., Ltd, Beijing, China). The shape, surface morphology, and material dimensions were characterized by thermal field emission scanning electron microscopy (SEM, Sigma 500 VP, Germany). The infrared absorption spectra were obtained by the Fourier transform infrared spectroscopy (FTIR, Nicolet iS5, Thermo, America) in the spectral range of 400–4000  $\text{cm}^{-1}$ . DOC contained in biochar was determined by the total organic carbon analyzer (Metash TOC 5000, Shanghai Metash Instruments Co., Ltd., Shanghai, China) with reference to sequencing extraction method.

Determination of basic physical and chemical properties of soil: Soil pH and SOM were determined by referring to Soil Agro-chemical Analysis [27]. The Cd content in different chemical forms in the soil was determined by the BCR (European Community Bureau of Reference) continuous extraction method, with the extraction order being weak acid extraction, reducible extraction, and oxidizable extraction. The extracted soil residue was digested using the  $\text{HNO}_3\text{--HClO}_4$  microwave digestion method. The total content of heavy metals and the content of each Cd form in the soil were determined by the graphite furnace atomic absorption spectrometry (iCE3000, Thermo Scientific, USA).

Statistical Analysis: The Origin Pro 2021b SPSS 21.0 software was used for statistical analysis, whereas the SPSS 21.0 software was used for correlation and significant analysis.

### 3. Result

#### 3.1. Characterization of biochar

The biochar characterization provided approaches for analyzing the Cd remediation mechanism of CsBC300 and CsBC600 in alkaline soil.

As can be seen from Fig. 1, the biochars prepared at different temperatures all had abundant pores. CsBC300 (a) was dominated by a sheet structure with disordered pore size distribution, while CsBC600 (b) has a more regularly shaped structure with a more uniformly distributed and abundant internal pore sizes.

As shown in Table 1, with the increase in the pyrolysis temperature, the ash content and pH of biochar increased, which is due to the large loss of oxygen and hydrogen, the increase of base cations and carbonate contents, and the disappearance of acidic functional groups [28]. Meanwhile, the specific surface area also increased with the increase in pyrolysis temperature, due to the breakdown of aliphatic alkyl and ester groups and the exposure of the aromatic lignin nuclei [29]. The H/C ratio reflects the aromaticity of the material; the O/C ratio reflects the existence of polar functional groups on the biochar surface that determines its hydrophilicity and hydrophobicity to a certain extent; and the (O + N)/C ratio represents polarity. The smaller the H/C ratio, the higher was the aromaticity. The higher the O/C ratio, better was the hydrophilicity. Finally, greater the (O + N)/C ratio, greater was the polarity [30, 31]. Therefore, although the aromaticity and hydrophobicity of CsBC600 was higher than CsBC300, it was less polar.

According to the infrared spectra of CsBC300 and CsBC600 (Fig. 2), both biochars contained different functional groups, like stretching vibration with a broad hydroxyl peak around 3400  $\text{cm}^{-1}$  [32,33]. The stretching vibration peak of aliphatic  $\text{CH}_2$  was around 2925  $\text{cm}^{-1}$  [34,35]. The absorption peak at 631  $\text{cm}^{-1}$  is the stretching vibration of the absorption peak of amide carbonyl, while the bending vibration peak at 1450–1400  $\text{cm}^{-1}$  was O–H, which can be used to determine whether there are carboxylic acid-containing compounds [36]. It was found that CsBC300 contained more oxygen-containing functional groups, while CsBC600 had more conjugated structures and stronger aromaticity, which was consistent with the results in Table 1.

#### 3.2. Effects of biochar on soil pH and organic matter

The soil pH and SOM directly affected the fractions of heavy metals. The result showed that after the Cd-contaminated soil was

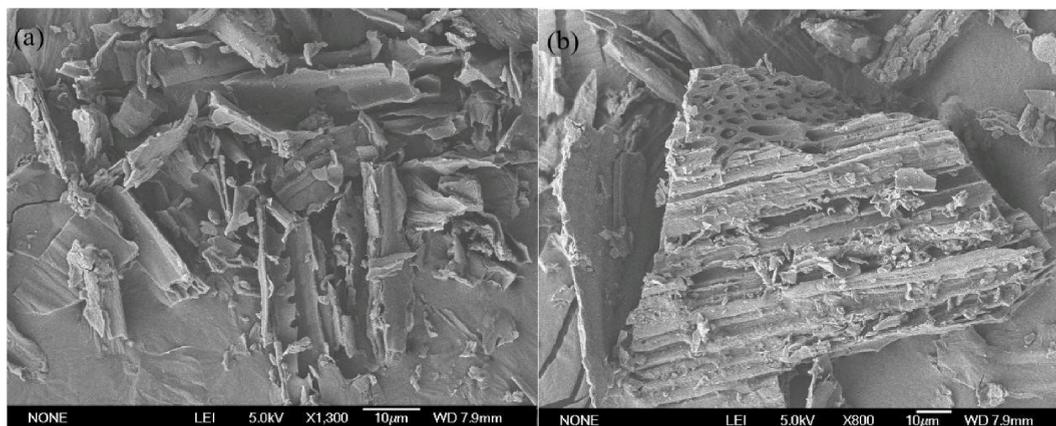
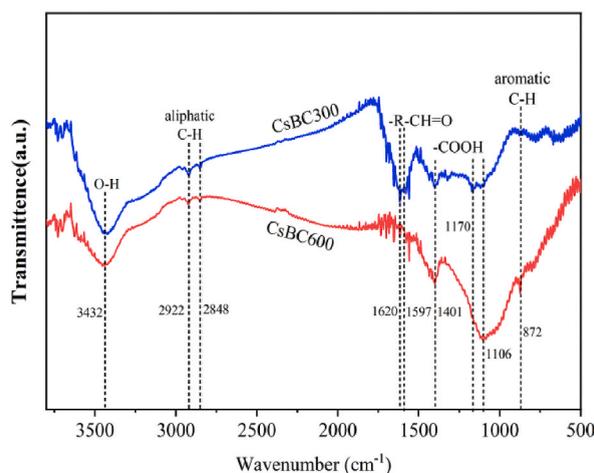


Fig. 1. SEM images of CsBC300 (a) and CsBC600 (b).

**Table 1**  
Physicochemical properties of CsBC300 and CsBC600.

Samples	Content (%)					DOC	H/C	O/C	(O + N)/C	Surface area (m <sup>2</sup> g <sup>-1</sup> )	Pore volume cm <sup>3</sup> g <sup>-1</sup>	pH
	C	H	O	N	Ash							
CsBC300	62.57	4.26	28.63	1.92	2.62	70.36	0.068	0.46	0.49	2.91	0.03	7.9
CsBC600	81.55	2.09	10.33	1.12	4.91	53.81	0.026	0.13	0.14	147.22	0.08	10.5



**Fig. 2.** FTIR spectrograms of CsBC300 and CsBC600.

treated with CsBC300 and CsBC600, the soil pH showed a downward trend, while the SOM showed an upward trend (Fig. 3).

It can be seen from Fig. 3(A) that within 10 days post the CsBC300 treatment, the soil pH showed a rapid downward trajectory, and with the increasing CsBC300 addition, the pH decreased even more, with the maximum (7.0%) decrease being 0.5 units. Within 10–30 d of soil incubation, the soil pH increased slightly. Subsequently, the soil pH in all the treatment groups decreased slightly and then stabilized at ~8.7 on the 120th day. This result confirmed that CsBC300 could effectively reduce the soil pH and was suitable for Cd-contaminated alkaline soil, which was consistent with a previous study [37].

As shown in Fig. 3(B), the soil pH increased by 0.18, 0.23, and 0.21 units, respectively, in the first 5 days of soil culture which treated with CsBC600 (3.0–7.0%). Subsequently, the soil pH of each group with the CsBC600 treatment gradually decreased and finally returned very close to the original soil pH on the 120th day.

As shown in Fig. 3(C-D) and Table S1, after treatment with CsBC300, the SOM content significantly increased with time and the treatment concentration, and showed a positive proportion, which was consistent with the previous study [38]. After treatment with CsBC600, the SOM content did not change significantly (Table 2). When the soil was cultured to 120 days, the SOM content slightly increased (10.08–23.80%). As compared with CsBC300, CsBC600 had very weak capability to increase SOM, which might be due to the decrease of the DOC content caused by high-temperature carbonization.

### 3.3. Effects of biochar on the transformation of Cd-fractions

The fractions of heavy metals in the soils near the mine directly reflect its mobility and bioavailability. The results showed that the Cd fractions in the soil periodically changed post addition of CsBC300 and CsBC600 to soil (Fig. 4). In the first 20 days of the soil culture, the Fr.1 (weak acid extractable fraction) and Fr.2 (reducible fraction) contents in the CsBC300 group decreased by 38.24–62.93% and 18.73–44.91%, respectively, while those in the CsBC600 group decreased by 28.85–40.56% and 12.573–34.85%, respectively. In contrast, the Fr.3 (oxidizable fraction) content in the CsBC300 and CsBC600 groups increased by 19.86–38.34% and 126.62–153.74%, respectively, but the increase in the CsBC600-treated soil was significantly higher than that in the CsBC300-treated soil. The Fr.4 (residual fraction) content increased by 41.00–48.20% in the CsBC300 group, while it decreased by 5.54–22.14% in the CsBC600 group. This was similar to a previous study [8,9] which indicated that high-temperature biochar can dissolve the stable Cd to some extent.

### 3.4. Correlation analysis

The correlation analysis (Pearson) between the Cd-fractions content, soil physicochemical properties, biochar preparation temperature and dosage, were shown in Table 3. The results showed that soil pH has a significant positive correlation with the biochar preparation temperature. Furthermore, the SOM content was significantly positively correlated with the remediation time and biochar

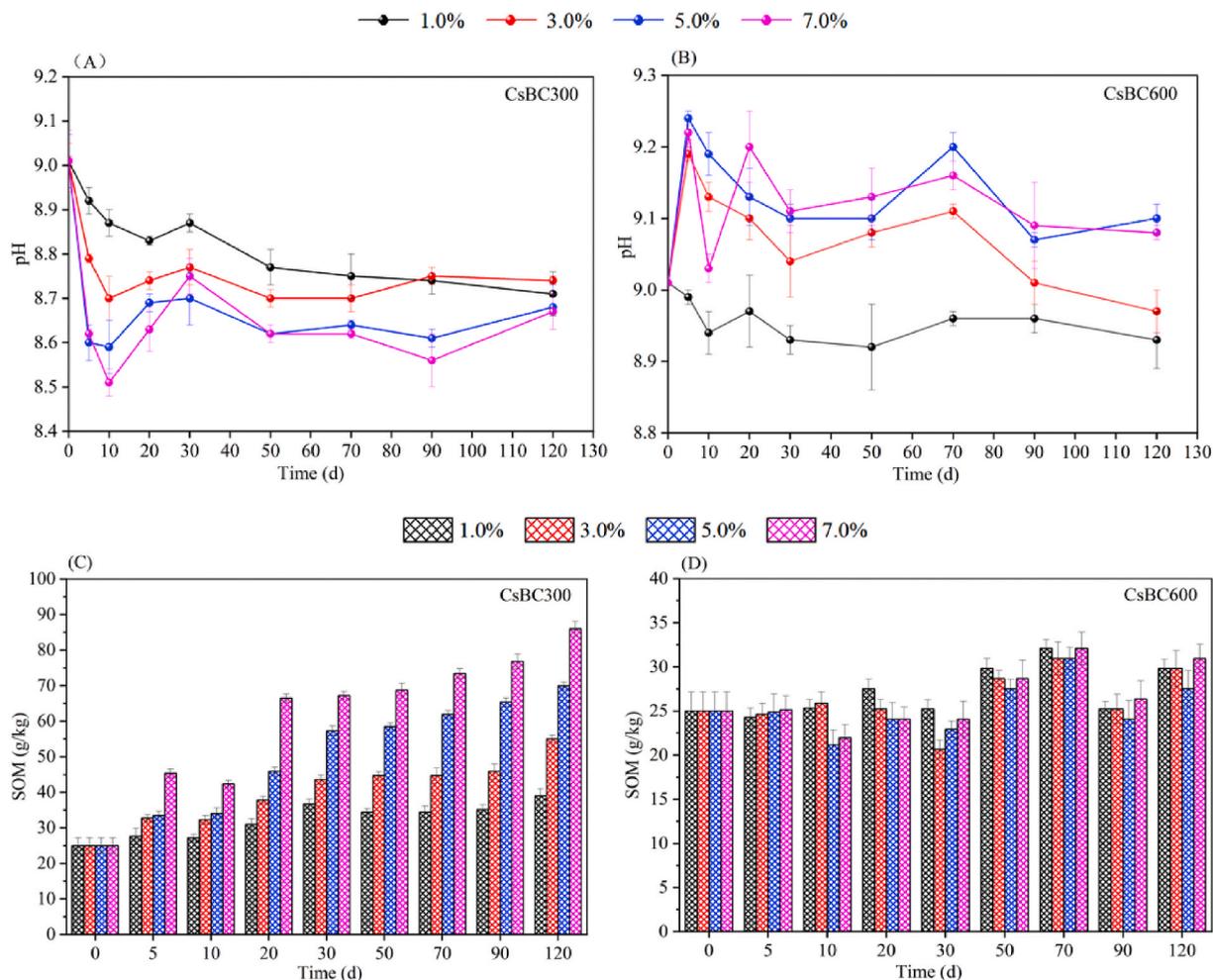


Fig. 3. Effect of biochar additions on the soil pH and SOM.

Table 2

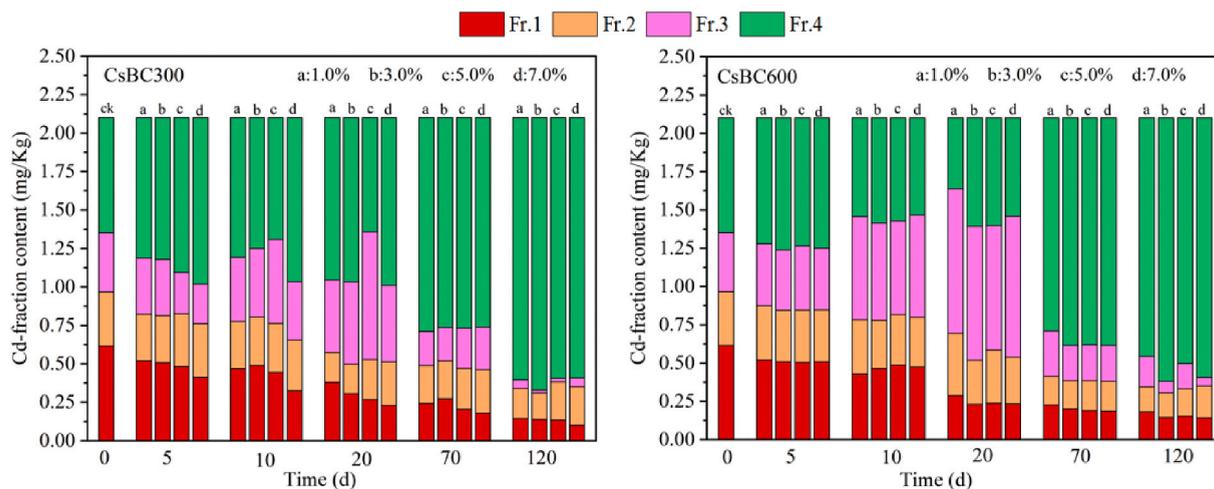
The significant difference analysis of SOM.

Time (d)	300 °C Biochar				600 °C Biochar			
	1%	3%	5%	7%	1%	3%	5%	7%
0	a	a	a	a	ab	ab	ab	ab
5	a	b	a	b	a	ab	ab	ab
10	a	b	a	b	ab	ab	a	a
20	ab	bc	b	c	ab	ab	ab	ab
30	bc	c	c	c	ab	a	ab	ab
50	bc	c	c	cd	ab	ab	ab	ab
70	bc	c	cd	cd	b	b	b	b
90	bc	c	cd	d	ab	ab	ab	ab
120	c	d	d	e	ab	ab	ab	ab

dosage, while being significantly negatively correlated with biochar preparation temperature. The weak acid extracted-Cd, reducible-Cd, and oxidizable-Cd contents in the soil showed significant negative correlation with the remediation time and SOM content, while the residual-Cd content was significantly positively correlated with the remediation time and SOM content.

4. Discussion

Biochar has been reported to have many significant effects on the soil properties [39,40], among which soil pH was the key geochemical factor that determined the bioavailability of heavy metals and their transport into the plant roots [41,42].



**Fig. 4.** Influence of biochar additions on Cd-fractions content ((Fr.1, Fr.2, Fr.3, and Fr.4 were weak acid extractable, reducible, oxidizable and residual fraction).

**Table 3**

The correlation between the Cd-fractions content and the impact conditions.

	Time (d)	Preparation temperature (°C)	Dosage (%)	pH	SOM
pH	-0.166	0.828**	-0.014	1	-0.754**
SOM	0.386**	-0.631**	0.338**	-0.754**	1
Fr.1	-0.841**	0.008	-0.108	0.230	-0.554**
Fr.2	-0.814**	0.031	0.111	0.108	-0.298*
Fr.3	-0.656**	0.249	-0.021	0.260	-0.403**
Fr.4	0.926**	-0.161	0.042	-0.282	0.550**

Note: \*\*. Correlation significant at 0.01 level (two-tailed). \*. Correlation significant at 0.05 level (two-tailed).

The results found that CsBC300 could reduce the soil pH within the experimental period. The main reasons were: 1) CsBC300 was the low-temperature pyrolyzed biochar, which was rich in organic matter and has rapid mineralization and oxidation reaction, thus promoting the SOM oxidation [43] and producing acidic substances [44]; 2) The pH of the CsBC300-treated soil was lower than that of the initial Cd-contaminated soil, and it neutralized and diluted the alkaline substances in the soil; 3) CsBC300 is rich in calcium carbonate salts and exchangeable cations [45], with the exchangeable cations react with the soil carbonate to form slightly soluble carbonate, which can then inhibit the carbonate hydrolysis and reduce the hydroxyl content in the soil [37]. Within 10–30 d of soil incubation, the soil pH increased slightly, because after the above acidification reaction reached equilibrium, the soil alkalization process gradually dominated since the role of functional groups, such as  $-\text{COO}^-$  and  $-\text{O}^-$  and the  $\pi$ -electron was carried by CsBC300 [46].

In contrast, CSBC600 increased the soil pH in the first 5 days. This may because: 1) CsBC600 is the high-temperature pyrolyzed biochar, which has a higher carbonization degree and ash content than CsBC300, making the CsBC600-treated soil pH much higher than that of initial Cd-contaminated soil since the lime effect. 2) The high-temperature pyrolysis conditions resulted in low microbial oxidation and mineralization rates of CsBC600, which was reflected by the high environmental stability. Therefore, it hardly provides acidic carboxyl functional groups for the soil, and therefore could not promote the SOM decomposition to produce acidic substances. 3) It can be explained by the previously reported biochar environmental recalcitrance index [47] that showed that the class B biochar (CsBC600) has more higher environmental stability and stronger aromaticity than the class C biochar (CsBC300) [48]. Therefore, the aromatic oxygen-containing functional groups carried by class B biochar could increase the soil pH. With the extension of soil culture time, the soil pH of each CsBC600 treatment group gradually decreased and finally returned close to the original soil pH on the 120th day. This is because that the aromatic oxygen-containing functional groups on biochar have been almost completely consumed, but the acid production by the SOM oxidation and biochar mineralization and oxidation reactions still continued, thus decreasing the soil pH [49].

In this study, our findings indicated that CsBC300 was more effective than the same dose of CsBC600 for the remediation of the Cd-contaminated alkaline wheat field soil. The reason may be related to the functional groups of biochar.

First, although the physical structure (decreased SSA and total pore volumes) of CsBC300 was not more favorable for Cd binding than that of CsBC600, elemental analysis showed that the CsBC300 biochar exhibited a higher O/C ratio (0.34) than CsBC600 (0.1). The FTIR spectrograms showed that CsBC300 contained more oxygen-containing functional groups. Liu et al. [49] proposed that the Cd adsorption by biochar was positively correlated with the O/C ratio, with higher O/C ratios indicating the presence of more O-containing functional groups that can form complexes with Cd. Thus, CsBC300 may have a strong Cd binding capacity when

introduced into the soil.

Second, as compared with CsBC600, CsBC300 had a greater ability to increase SOM. Numerous studies have shown that biochar can rapidly increase SOM content [50,51], but the effect on the SOM content was also related to the application amount and stability of biochar. Quan et al. [52] found that the soil organic carbon content increased after the biochar application, because it increased the soil carbonate component, while also providing more adsorption sites with large specific surface area and high porosity. In this way, soil carbon sequestration and nutrient retention can be improved. [53] showed that the rice stalk biochar could significantly increase the SOM content, as it was positively correlated with the residual Cd content change.

In this study, the mobility factor (MF) [54] was used to further evaluate the potential movement of Cd in the soil. The MF of Cd decreased significantly after the contaminated soil was treated with CsBC300 and CsBC600 and was only 0.06 and 0.07 on the 120th day, respectively. Studies have shown that biochar can significantly reduce the weak acid extracted-Cd content and increase the residual-Cd content of, which may be because biochar has a loose porous structure, large specific surface area, and rich oxygen-containing functional groups, allowing it to bind heavy metals via electrostatic adsorption, ion exchange, and complexation and chelation of the functional groups [55-57]. Therefore, the study results were consistent with these conclusions, thereby indicating that the acid-extracted Cd content effectively reduced after the Cd-contaminated soil culture was treated with CsBC300 and CsBC600 for 20 days, ultimately reducing the Cd-induced toxicity.

## 5. Conclusion

Biochar has a certain remediation effect on the Cd-contaminated alkaline wheat field soil, which reduced the Cd availability and improved the soil quality. The main conclusions are:

- (1) The physicochemical properties, specific surface area, functional groups, and pore structure of biochar differed with the different preparation temperatures.
- (2) Both CsBC300 and CsBC600 had good remediation effects on the Cd-contaminated alkaline wheat field soil by significantly reducing the acid-extracted Cd content and increasing the residual Cd content, thereby facilitating the transformation of Cd from an unstable to a stable state
- (3) CsBC300 could significantly reduce the soil pH and increase the SOM content. Higher the dosage, greater was this effect. However, the effect of CsBC600 on soil pH and SOM was not significant.

## 6. Research prospects

Focusing on the treatment of Cd-contaminated alkaline soil in the wheat fields in northern China, this study not only prepared and characterized biochar at different temperatures and pot remediation experiments, but also discussed how biochar regulated the Cd bioavailability in the soil and how it affected the soil's physico chemical properties. The viewpoints mentioned in this study valuable for using biochar in the remediation of the Cd-contaminated alkaline soil in the wheat fields in northern China. However, there are still some shortcomings in this study:

- (1) Since the redox potential of soil and the change of soil fertility have not been investigated in this study, the regulation mechanism of organisms on the Cd bioavailability in the soil needs further research.
- (2) This study did not evaluate the effect of the structure and composition of soil microorganisms, which has certain limitations.

## Author contribution statement

Meizhen Tang; Lianglun Sun: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

Guoquan Zhang: Conceived and designed the experiments; Performed the experiments; Contributed reagents, materials, analysis tools or data.

Xinyu Li; Xinyu Zhang; Wei Hang; Yan Gao: Conceived and designed the experiments.

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## Data availability statement

Data included in article/supp. material/referenced in article.

## Declaration of interest's statement

The authors declare no conflict of interest.

## Appendix A. Supplementary data

Supplementary data related to this article can be found at <https://doi.org/10.1016/j.heliyon.2023.e12949>. [58].

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